Considerations for Environmental TEM in catalysis and nanoparticle research

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Transmission electron microscopy (TEM) is used extensively in catalysis research. Recent developments in instrumentation include monochromation of the electron source and aberration correction of both the condenser and the objective lens systems. These developments are now also being introduced onto the environmental TEM (ETEM). The improved spatial resolution and interpretability provided by these additions are beneficial for imaging the surface structures and dynamics of catalyst nanoparticles. These developments also provides exciting new possibilities for investigating chemical reactions and understanding both the interaction of fast electrons with gas molecules and the effect of the presence of gas on high-resolution imaging.

Using an FEI Titan ETEM equipped with a monochromator and an aberration corrector on the objective lens [1], we have investigated Au/graphene samples in vacuum and in a hydrogen atmosphere at pressures up to 700Pa [2]. The gases were introduced into the environmental cell using digitally controlled mass flow controllers, providing accurate and stable control of the pressure in the cell. The effects on high resolution imaging were investigated by imaging gold nanoparticles below 5nm in diameter (see Figure 1 left and middle).

State-of-the-art aberration corrected TEMs provide electron micrographs with high spatial resolution encouraging microscopists to analyze data more quantitatively. A gaseous atmosphere in the pole-piece gap of the objective lens of the microscope alters both the incoming electron wave prior to interaction with the sample and the outgoing wave below the sample [3]. Whereas conventional TEM samples are usually thin (below 10-20 nm), the gas in the environmental cell fills the entire gap between the pole pieces and is thus not spatially localized. We have investigated the effects on imaging and spectroscopy capabilities of the instrument caused by the presence of the gas around the sample. The loss of beam intensity when traversing the pole piece gap was measured by recording the signal outside the sample region on the pre-GIF CCD camera (see Figure 1 right).

Results from imaging in various elemental as well as di-molecular gases and their effect on imaging and spectroscopy in the environmental transmission electron microscope as well as other examples from catalysis studies will be presented.

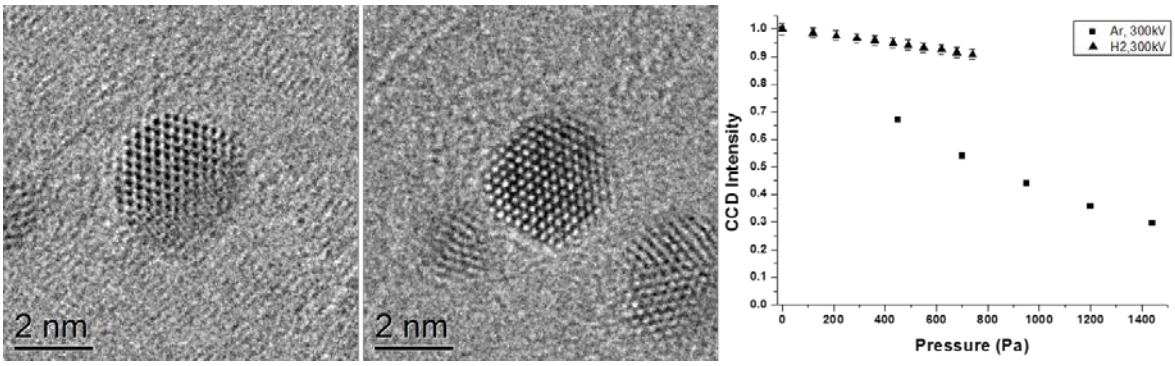

Figure 1: Left: Au nanoparticle imaged in vacuum; middle: graphene-supported Au nanoparticle imaged in hydrogen at 200Pa; right: Variation in intensity measured on the CCD as a function of gas pressure in the sample region.

[1] Hansen, T. W., Wagner, J. B. and Dunin-Borkowski, R. E., *Mater. Sci Technol.* **26**, 1338 (2010).

[2] Hansen, T. W. and Wagner, J. B., *Microsc. Microanal.*, in press.

[3] Wagner, J. B. *et al.*, *Micron*, in press.